IS 494 (1970): aluminium stearate for lubricants [CHD 1: Inorganic Chemicals]

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SPECIFICATION FOR ALUMINIUM STEARATE FOR LUBRICANTS

(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3 *August* **1970**

Indian Standard

SPECIFICATION FOR ALUMINIUM STEARATE FOR LUBRICANTS

(First Revision)

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Indian Standard

SPECIFICATION FOR ALUMINIUM STEARATE FOR LUBRICANTS

(First Revision)

0 FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 25 March 1970, after the draft finalized by the Lubricants and Related Products Sectional Committee had been approved by the Chemical Division Council and the Mechanical Engineering Division Council.

0.2 This standard was first issued in 1955 and amended in 1962, in order to ensure the manufacture and supply of material of a quality suitable for use in the manufacture of lubricating greases. In this revision the requirements for ash have been substituted by a requirement for aluminium stearate, and the methods of test modified and brought up to-date. The method of determination of worked penetration has also been improved in the light of experience.

0.3 It is known that aluminium stearate can be used as an emulsifier in some cosmetic preparations, but this application is not yet established in this country. As such the cosmetic grade aluminium stearate is not covered in this standard. This position will, however, be reviewed when there is a demand from the cosmetic industry.

0.4 This standard contains clauses **3.1** and **3.3.2** which call for agreement between the purchaser and the supplier.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for aluminium stearate intended primarily for use in the manufacture of lubricating greases.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall consist essentially of aluminium stearate of high gel forming property and shall be in the form of a fine, dry powder, white or nearly white in colour.

2.2 Fineness — The material shall comply with the following requirements:

Remaining on 212-micron IS Sieve *(see* IS : 460-1962*), percent by weight, *Max* 3

Remaining on 63-micron IS Sieve (*see* IS : 460-1962*), percent by weight, *Max* 50

2.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. References to the relevant clauses of Appendix A are given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ALUMINIUM STEARATE FOR LUBRICANTS

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Moisture content, percent by weight, *Max*	1.5	A-2
ii)	Inorgantic acidity or alkalinity	Shall pass the test	A-3
iii)	Acidity of acetone extractabtes [as $CH_3(CH_2)_{16}COOH$], percent by weight, *Max*	10.0	A-4
iv)	Aluminium stearate content (as Al_2O_3), percent by weight, *Min*	10.0	A-5
v)	Iron content (as Fe_2O_3) percent by weight, *Max*	0.10	A-6
vi)	Dispersion in benzene	Shall pass the test	A-7
vii)	Worked penetration of dispersed material, in 0.1 mm, *Max*	300	A-8

3. PACKING AND MARKING

3.1 The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

*Specification for test sieves (*revised*).

3.2 The container shall be securely closed and marked with the name of the manufacturer and the registered trade-mark, if any; name of the material and its weight; and an identification in code or otherwise to enable the lot or consignment of manufacture to be traced back from records.

3.2.1 The product may also be marked with Standard mark.

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3, *and Table* 1)

METHODS OF TEST FOR ALUMINIUM STEARATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals shall be employed in the tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MOISTURE CONTENT

A-2.1 Procedure — Weigh accurately about 1 g of the material in a squat type glass weighing bottle (*see* IS : 1574-1960†), 50 mm in diameter

*Specification for water, distilled quality (*revised) (* Since revised)

†Specification for glass weighing bottles.

and 25 mm in height, fitted with a ground glass stopper. Remove the stopper, and place both the stopper and bottle separately in an air-oven maintained at 105° ± 2°C. After three hours of heating, replace the stopper in the bottle while still in the oven and cool in a desiccator before weighing. Remove and replace the stopper quickly to equalize pressure. Calculate the loss in weight as percentage of the weight of the material taken for the test.

A-3. TEST FOR INORGANIC ACIDITY OR ALKALINITY

A-3.1 Reagents

A-3.1.1 *Rectified Spirit* — conforming to IS : 323-1959*.

A-3.1.2 *Methyl Orange Indicator* — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-3.1.3 *Bromocresol Purple Indicator* — Warm 0.1 g of bromocresol purple with 5 ml of rectified spirit until dissolved. Dilute with 100 ml of ethyl alcohol (20 percent by volume), add 3.7 ml of 0.05 N sodium hydroxide solution and sufficient ethyl alcohol (20 percent by volume) to produce 250 ml.

A-3.2 Procedure — Weigh about 5 g of the material and add 10 ml of rectified spirit. Shake vigorously for one hour with 90 ml of freshly boiled water at room temperature. Filter and test the filtrate for acidity and alkalinity, using methyl orange and bromocresol purple indicators, respectively.

A-3.2.1 The material shall be taken to have passed the test if neither the methyl orange nor the bromocresol purple decolorizes.

A-4. DETERMINATION OF ACIDITY OF ACETONE EXTRACTABLES

A-4.1 Regents

A-4.1.1 *Acetone*

A-4.1.2 *Standard Alcoholic Potassium Hydroxide Solution* — 0.1 N.

A-4.1.3 *Phenolphthalien Indicator* — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit that has previously been neutralized to the indicator.

A-4.2 Procedure — Weigh accurately about 10 g of the material into a 250-ml conical flask. Add 50 ml of acetone and then reflux the contents of the conical flask for 10 minutes from the start of the boiling of the solvent keeping the conical flask on a low-heated hot-plate and then remove the conical flask and carefully filter the contents of the conical

*Specification for rectified spirit (*revised*).

flask through a suitable filter paper, collecting the filtrate in a clean flask. Wash the aluminium stearate with another 50-ml portion of acetone collecting the washings into the flask containing the filtrate. Titrate the filtrate and the washings against standard alcoholic potassium hydroxide solution using phenolphthalein indicator.

A-4.3 Calculation

Acidity of acetone extractables [as $CH_3(CH_2)_{16}COOH$], percent by weight $= \frac{28{\cdot}45\,VN}{W}$

where

V = volume in ml of standard alcoholic potassium hydroxide solution,

N = normality of standard alcoholic potassium hydroxide solution, and

W = weight in g of the material taken for the test.

A-5. DETERMINATION OF ALUMINIUM STEARATE CONTENT

A-5.0 Aluminium stearate content is estimated as Al_2O_3.

A-5.1 Reagents

A-5.1.1 *Sodium Bisulphate*

A-5.1.2 *Dilute Hydrochloric Acid* — 5 N.

A-5.1.3 *Ammonium Chloride*

A-5.1.4 *Concentrated Nitric Acid — see* IS : 264-1950*.

A-5.1.5 *Dilute Ammonium Hydroxide* — 4 N.

A-5.1.6 *Methyl Red Indicator* — Dissolve 0.15 g of methyl red in 500 ml of water.

A-5.1.7 *Ammonium Nitrate Solution* — Approximately 2 percent *(w/v).*

A-5.2 Procedure — Weigh accurately about 1 g of the material and ignite at a dull red heat until all carbonaceous matter has been burnt off. Cool and then fuse with the requisite amount of sodium bisulphate in order to render the whole ash soluble in dilute hydrochloric acid. Dissolve the melt in dilute hydrochloric acid, filter and wash the residue on the filter paper thoroughly with water. Add 5 g of ammonium chloride and 2 drops of concentrated nitric acid to the filtrate and washings; heat to boiling and add dilute ammonium hydroxide, drop by drop, until the solution smells

*Specification for nitric acid. (Since revised).

faintly of ammonia and shows distinct yellow colour on the addition of two drops of methyl red indicator. Boil for 2 minutes, filter immediately through a suitable filter paper (Whatman No. 41 or equivalent) and wash the residue with hot ammonium nitrate solution till the filtrate is free from chlorides. Dry the residue and ignite to constant weight at 1 000° to 1 200°C in a tared porcelain or a silica crucible.

A-5.3 Calculation — Calculate the aluminium stearate expressed as aluminium oxide as follows:

$$\text{Aluminium stearate (as } Al_2O_3\text{), percent by weight} = \frac{100\,w}{W} - 1{\cdot}4297\,A$$

where

w = weight in g of the ignited residue ($Al_2O_3 + Fe_2O_3$),

W = weight in g of the sample taken for the test, and

A = percent by weight of iron as determined in **A-6.**

A-6. DETERMINATION OF IRON CONTENT

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinder* — of 100 ml capacity having a mark at 50 ml (*see* IS : 4161-1967*).

A-6.2 Reagents

A-6.2.1 *Sodium Bisulphale*

A-6.2.2 *Dilute Hydrochloric Acid* — approximately 5 N.

A-6.2.3 *Citric Acid Solution* — approximately 10 percent *(w/v).*

A-6.2.4 *Thioglycollic Acid*

A-6.2.5 *Concentrated Ammonium Hydroxide Solution* — sp gr 0.90.

A-6.2.6 *Standard Iron Solution* — Dissolve 0.604 0 g of ferric ammonium sulphate [$FeNH_4(SO_4)_2$, 12 H_2O] in 10 ml of dilute sulphuric acid (10 percent) and dilute with water to 1 000 ml. One millilitre of this solution is equivalent to 0.1 mg of iron (Fe_2O_3).

A-6.3 Procedure

A-6.3.1 Weigh accurately about 1 g of the material and ignite at a dull red heat until all carbonaceous matter has been burnt off. Cool and then fuse with requisite amount of sodium bisulphate in order to render the whole ash soluble in dilute hydrochloric acid. Dissolve the melt in dilute

*Specification for Nessler cylinders.

hydrochloric acid, filter and wash the residue on the filter paper thoroughly with water. Make up the volume of the filtrate to 1 000 ml with water. Transfer 10 ml of this solution to a Nessler cylinder and dilute to 50 ml.

NOTE — To carry out a blank test, dilute 3 ml of dilute hydrochloric acid to 50 ml; and 10 ml of this dilute acid to another Nessler cylinder and dilute to 50 ml.

A-6.3.2 To each Nessler cylinder, add 5 ml of the citric acid solution 3 drops of thioglycollic acid and ammonium hydroxide solution dropwise until the solution is alkaline. Allow to stand for 5 minutes for the full intensity of colour to develop in the test solution.

A-6.3.3 To obtain an approximate match, add standard iron solution dropwise from a semi-micro-burette to the blank, until the intensity of colour obtained matches with that produced in the test solution (allowing time after each addition for the complete development of colour). Note the volume of standard iron solution added. Carry out another blank test adding the noted volume of standard iron solution in one operation. If the colour obtained does not match with that of the test solution, repeat this procedure and blank solutions until the colours match on fresh test.

A-6.4 Calculation — Calculate the iron content expressed as iron oxide as follows:

$$\text{Iron (as } Fe_2O_3\text{), percent by weight} = \frac{V}{W}$$

where

V = volume in ml of standard iron solution required in the blank, and

W = weight in g of the material taken for the test.

A-7. TEST FOR DISPERSION IN BENZENE

A-7.1 Procedure — Heat a weighed amount of the material with four times its weight of benzene and allow it to stand overnight. Examine the solution on the next day.

A-7.2 The material shall pass the test if it is completely soluble in benzene and forms a clear gel.

A-8. DETERMINATION OF WORKED PENETRATION OF DISPERSED MATERIAL

A-8.1 Procedure — Prepare a 10 percent dispersion of the material in an enamel panel by adding the aluminium stearate powder gradually with stirring to a naphthenic mineral oil having a viscosity of 19.5 to 22.8 centistokes at 38°C. Continue stirring at room temperature till a slurry

is produced. Heat the mixture on an electric hot-plate to 150°C with constant rapid hand agitation. Withdraw the liquid grease so formed from the oven and immediately pour it into a flat 500-ml can, filling the can to the brim. Place it in an electric oven maintained at 120°C. As soon as the liquid grease has attained the temperature of 120°C, cut off the source of the heat and allow both the oven and the liquid grease to cool overnight to any definite temperature below 25°C Determine the worked penetration of the dispersed material (60 strokes) according to P:60 (1967) of IS : 1448*.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF ALUMINIUM STEARATE FOR LUBRICANTS

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry when used.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable clean, dry and air-tight metal or glass containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each simple container shall be sealed air-tight by suitable means after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

*Methods of test for petroleum and its products: consistency of lubricating grease by cone penetrometer (Since revised).

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers *(n)* to be chosen from the lot shall depend on the size of the lot *(N)* and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE	SAMPLE SIZE
N	*n*
(1)	(2)
3 to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 and over	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, random number tablet shall be used; in case such tables are not available, the following procedure may be adopted:

Starting from any container, count them as 1, 2, 3....., *r* and so on in one order, where *r* is the integral part of *N/n*. Every *r*th container thus counted shall be withdrawn from the lot.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw, with an appropriate sampling instrument, small portions of the material from different parts of each container selected for sampling and freshly opened. The total quantity of the material drawn from each container shall be sufficient to conduct tests for the characteristics given in **2** and shall not be less than 300 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, an equal quantity shall be taken from

each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 0.5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for the referee.

B-3.1.3 The remaining portions of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 40 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7.** The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, one to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of a composite sample (**B-3.1.2**) and a set of individual samples (**B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for moisture content and aluminium stearate content shall be done on each of the individual samples.

B-4.2 Test for all the other characteristics given under **2** shall be done on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — For each of the characteristics tested on individual samples, the mean and the range shall be calculated as follows:

Mean ($\bar{X}$) = sum of test results divided by the number of test results

Range (R) = difference between the maximum and the minimum values of the test results.

B-5.1.1 The lot shall be declared to have satisfied the requirements for these characteristics if for each the value of the expression ($\bar{X} + 0.4\ R$) does not exceed the specified maximum limit.

B-5.2 For Composite Sample — The composite sample shall meet all the requirements tested on it.

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5-8-56C, L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	320 10 84
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